Introduction

Health is a basic consideration for all human beings and diseases must be healed in a minimum of time. Hence, man consumes huge numbers of drugs in order to battle against the diseases. Drugs not only cure the illnesses but also support to avoid the diseases [1-3]. Drugs are sold in numerous forms such as tablets, capsules, aerosols, powders and among all the forms tablet form is appropriate to use. Hormones are the chemical substance produced by the body that controls some of the functions of body. Hormones are one among the biomolecules utilized by the humankind due to their importance in their life. The lower and higher level of the hormones leads to many discomforts [4-5]. Some effects of the drugs and the hormones grab the attention of the analyst and hence pharmaceutical analysis gained an important role in the field of analytical chemistry. Therefore, it is necessary to analyse the concentration of the drugs and hormones.

Electroanalytical approaches are the main group of analytical chemistry. These methods have abundant advantages such as low-priced instruments, a lesser amount of solvents, high sensitivity, etc., compared to the other traditional analytical techniques such as chromatography, electrophoresis, spectrophotometry, etc. [6-8]. The electrochemical technique possesses the three main types of techniques such as Potentiometric, Coulometric, and voltammetric methods. Voltammetric are the most imperative technique because it measures both current and the potential. The voltammetry method was divided into various types. Cyclic voltammetry (CV) is one of the main classes of the voltammetric technique. CV measures the current when the potential is applied between the reference and the working electrode. In CV, the applied potential is the regulated factor, and it can be varied according to the redox property of the drugs and hormones being analysed [9-12]. CV is very popular technique extensively used worldwide to gain knowledge about diffusion or adsorption-controlled process at the electrode, concentration of analyte, electron transfer, kinetics of the transfer process, reaction mechanism, reaction intermediates, and especially to know the redox property of the electro active molecules [13].

Carbon paste electrode (CPE) is a homogenous mixture of the graphite powder, binder and it is compacted at the designed holder [14]. Traditional forms of the graphite moiety is substituted by the new carbon materials such as graphene is the two-dimensional honeycomb lattice arrangement of thin layer of the graphite and carbon nanotubes constitutes the rolled up graphene layers. The above all forms of the carbon materials has great benefits to select as sensing electrode such as low background current, electrochemically inert, easy-

to-prepare and chemical stability [15]. Electroanalysis of drugs and hormones with the CPE is very significant.

Even though the CPE detect the analyte but with modifier (adding foreign substances) CPE enhance the analytical signal of the targeted analyte. Hence, modification of the CPE plays a crucial role in the electrochemical analysis of the different molecules via voltammetry technique. Some of the modifications methods to modify the electrode are electropolymerisation and the immobilisation or casting technique. Electropolymerisation involves the polymerisation of monomer solution at the electrode by the controlled potential scanning provides the thin conducting layer on the electrode surface [16, 17]. Immobilisation method consists of drop coting the surfactant (surface-active agents such as sodium dodecyl sulphate, Cetyltriethylammonium bromide, TX-100 etc.) on the surface of the electrode [18]. Both modification methods are easy to fabricate the electrode and consume less time and solvents.

The voltammetric sensor based on the carbon material utilised for the detection of the drugs and hormones has many advantages. It provides high sensitivity, selectivity, stability and enhanced electrocatalytic activity towards the analysis of the drugs and hormones.

Summary of the Thesis

The main attention of the work presented in the thesis was development of modified carbon paste electrode for the detection of the drugs and hormones through the cyclic voltammetric technique. The pharmacokinetic and diagnostic pharmacology analysis in biological systems provides valuable information hence pharmaceutical analysis of the drugs and hormones are significant in the field of the analytical chemistry. The various allotropic forms of the carbon such as graphite, carbon nanotubes, graphene are utilized and the above all forms has some benefits such as high conductivity, chemically stable, low background current, etc. and hence carbon paste electrode are used as sensing material. The allotropic forms of carbon with the incorporated modifier enhance the sensing capability of the proposed sensor. The current research work illustrates the designed electrochemical sensor based on the carbon paste electrode was employed for the voltammetric detection of the some drugs molecules and the hormones. The targeted goal of the present thesis is to fabricate the electrochemical sensor based on the carbon paste electrode with modifier to detect the low concentration of the drugs and hormones with the application of the cyclic voltammetry. The author also has assumed that a contribution coming from our laboratories presented in this paper, may offer an interesting insight in to the electrochemistry field.

Organization of the Thesis

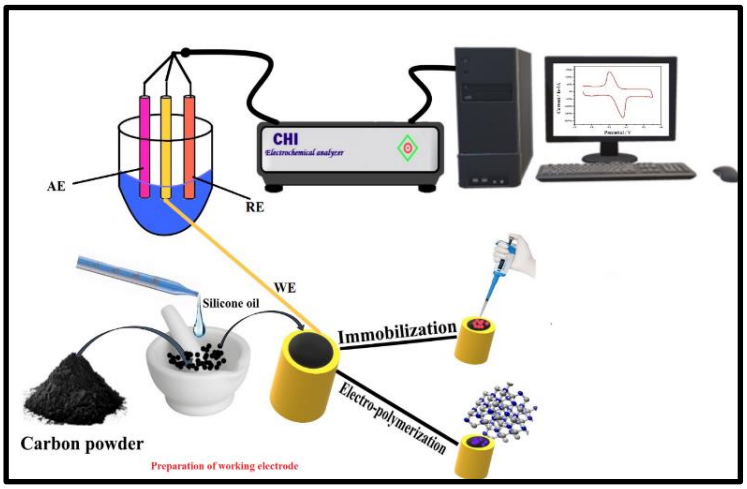
The thesis entitled "Mechanistic Investigation of Drugs and Hormones at Chemically Modified Carbon Paste Electrodes – A voltammetric study" has been outlined into following chapters.

Chapter 1: **Introduction, Review of cyclic voltammetry, and Theoretical Discussions**

This chapter covers introduction to drugs and hormones, electroanalytical methods, voltammetry techniques, Theoretical aspects and applications of cyclic voltammetry, different types of voltammetric technique, discussions about carbon paste electrode as sensing materials, different allotropic forms of the carbon, A brief literature review of voltammetric analysis of some drugs and hormones has been included. The main objectives of the research work are also incorporated.

Chapter 2: **Experimental**

This chapter provides the detailed information regarding the materials and methods utilized in the present research work. Electrochemical analyser used for recording the voltammograms, recording device, electrochemical cell, three electrode system, working electrode were described. The detailed procedure followed for the construction of bare carbon paste electrode and the modified electrode has been included.



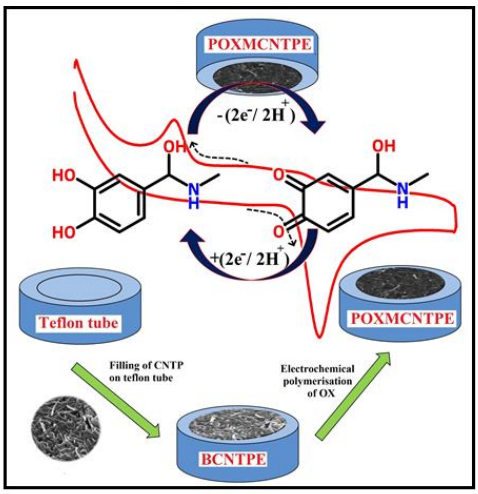
Chapter 3A: Electrochemical sensing of adrenaline using surface modified carbon nanotube paste electrode

(**Materials Chemistry and Physics,** 262, 2021, 124293

https://doi.org/10.1016/j.matchemphys.2021.124293, ISSN: 0254-0584, **I. F: 4.0**)

This chapter describes a new interesting insight into the construction of a voltammetric sensor for the detection of Adrenaline (AD) has been presented. Poly oxalic acid modified

carbon nanotube paste electrode (POXMCNTPE) exhibited the boosted detecting ability towards the AD compared to the bare CNTPE (BCNTPE) as a sensing device. The detection of AD (pH 6.5) has been executed through the cyclic voltammetry (CV) and differential pulse voltammetry (DPV) approaches. The surface morphological features, elemental analysis and electrochemical properties of the proposed electrode are revealed by Field emission scanning electron microscopy (FE-SEM), Energy dispersive X-ray spectroscopy (EDS), Electrochemical impedance spectroscopy (EIS), and CV technique. The detection limit (DL) 3.1×10^{-8} M and quantification limit (QL) 1.0×10^{-7} M was gained from the proposed method. Hence, the proposed procedure was effectively implemented to detect the AD in the injection sample and as well as in the blood serum sample.

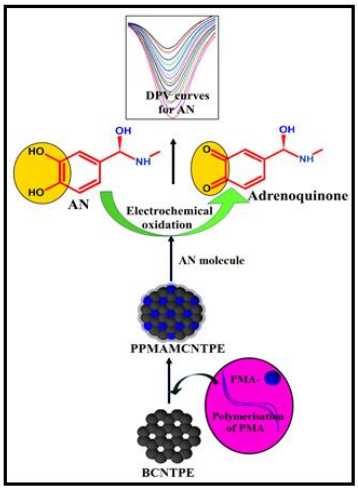


Chapter 3B: Polymerized Carbon Nanotube Paste Electrode as a Sensing Material for the Detection of Adrenaline with Folic acid

(**Monatshefte für Chemie - Chemical Monthly, 152, 2021, 44-420**

https://doi.org/10.1007/s00706-021-02756-0, ISSN: 0026-9247, **I. F: 1.45**)

This chapter covers a new intuition to construct the sensitive platform based on the Poly Phenylmethanoic acid modified carbon nanotube paste electrode (PPMAMCNTPE) to investigate the electroanalytical performance of Adrenaline (AN), which acts as both neurotransmitter and hormone. The PPMAMCNTPE reveals a high detectability than the bare carbon nanotube paste electrode (BCNTPE) through cyclic voltammetry technique (CV). The morphological and electrochemical conductivity of the electrode was characterized by (FE-SEM and EIS. The lowest detection limit (4.5×10^{-8} M) was gained within a linearity of the AN concentration range of 10 µM-1100 µM via differential pulse voltammetry (DPV) approach at the PPMAMCNTPE. Finally, the PPMAMCNTPE was effectively implemented for the voltammetric detection of AN in both injection and blood serum samples.

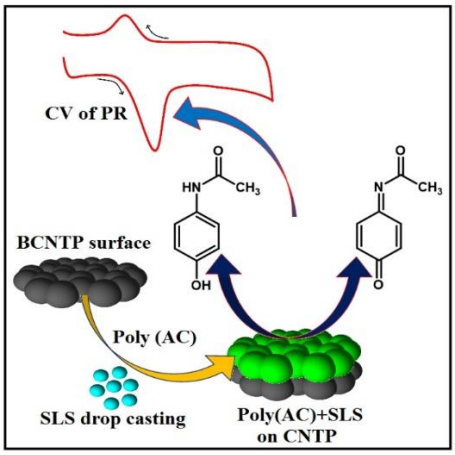


Chapter 4A: Electrochemical Sensing of Paracetamol Using Electropolymerised and Sodium Lauryl Sulfate Modified Carbon Nanotube Paste Electrode

(ChemistrySelect, 5, 2020, 9323-9329,

10.1002/slct.202002626, ISSN: 2365-6549. **I. F: 2.10**)

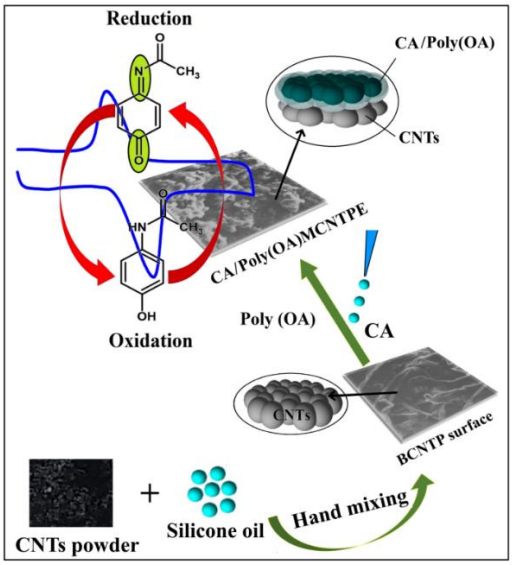
This chapter illustrates the sensing tool based on the electropolymerisation of alizarin carmine (AC) and immobilization of sodium lauryl sulfate (SLS) on the surface BCNTPE has been developed. Poly (AC) SLS modified CNTPE (PACSLSMCNTPE) facilitates the enhancement in voltammetric signal for PR in 0.2 M supporting electrolyte (pH 7.0). The surface morphology features of the proposed electrodes were gained from the FE-SEM. The calibration curve for PR concentration level from 4.0-100 μM was plotted and 0.06 μM limit of detection (LD) 0.21 μM limit of quantification (LQ) was attained. No major disturbance in the voltammetric signal for PR in the existence of dopamine (DA) and estriol (ES) discloses the selectivity of the proposed sensor. The prepared electrode holds an adequate repeatability, sufficient reproducibility, and good antifouling feature. The projected sensor was magnificently applied for the electroanalysis of PR in the tablet formulations.



Chapter 4B: Electroanalytical Determination of Paracetamol using polymerised Carbon Nanotube Based Sensor

(Accepted in Journal of Electronic Materials, ISSN: 0361-5235, **I. F. 1.93**)

Special attention focused on fabricating a sensitive voltammetric sensor based on Cetrimide (CA) incorporated on the poly Oxalic Acid modified carbon nanotube paste electrode (POAMCNTPE). The unmodified and modified electrodes topographical features and electrochemical characterisations were compared using a variable pressure scanning electron microscope (VP-SEM) and EIS. The scan rate study reveals that the redox reaction of the PC at the surface of the modified electrode was controlled by diffusion. The DL of 1.50×10^{-8} M and quantification limit (QL) of 5.02×10^{-8} M was gained by utilizing the (DPV. The practicability of the proposed electrode was examined to be successful towards the quantification of PC in both pharmaceutical and biological samples.



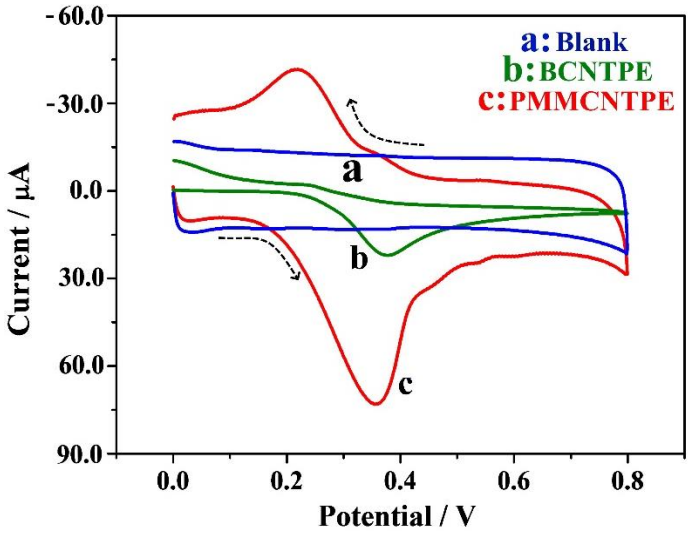
Chapter 5A: Enhanced voltammetric detection of Acetaminophen by using carbon nanotube modified electrode as an electrochemical sensor

(**Journal of Electrochemical Science Engineering,** 10, 2020, 29-40

http://dx.doi.org/10.5599/jese.717**,** ISSN:1847-9286, **CiteScore: 1.7**)

This chapter describes new aspects associated with electro-catalytic activity of poly (methyl orange) modified carbon nanotube paste electrode (PMMCNTPE) towards the detection of Acetaminophen (AP) which is typically used worldwide as a pain reliever, were explored through implementation of CV and DPV techniques. Bare carbon nanotube paste electrode (BCNTPE) was modified by methyl orange using the electro-polymerizing method. The morphological studies of the BCNTPE and PMMCNTPE was achieved with the FE-SEM

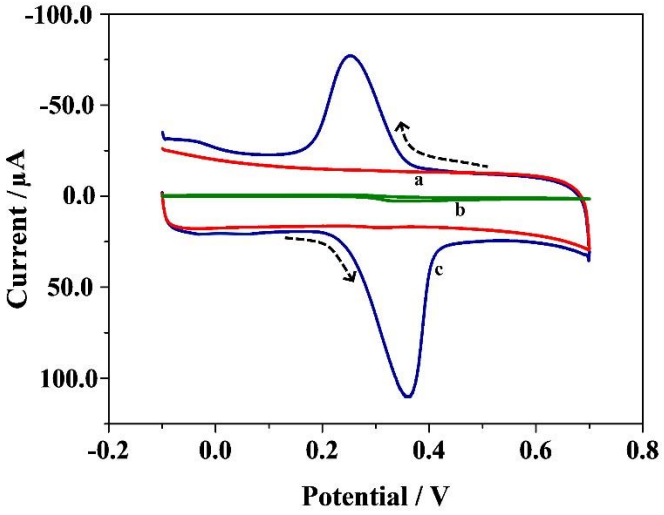
analysis. It was found that under optimized experimental conditions, PMMCNTPE imparts the analytical curve for AP with detection limit of 3.8×10^{-8} M and limit of quantification of 1.2×10^{-8} M. The proposed sensor exhibited acceptable reproducibility, admirable stability, and adequate repeatability. The feasibility of the constructed electrochemical sensor to detect AP was successfully tested in pharmaceutical formulations.



Chapter 5B: Electroanalytical Determination of Acetaminophen using Polymerized Carbon Nanocomposite based Sensor

(**Chemical Data Collections,** 33, 2021, 100718, https://doi.org/10.1016/j.cdc.2021.100718, ISSN: 24058300, **CiteScore: 1.2**)

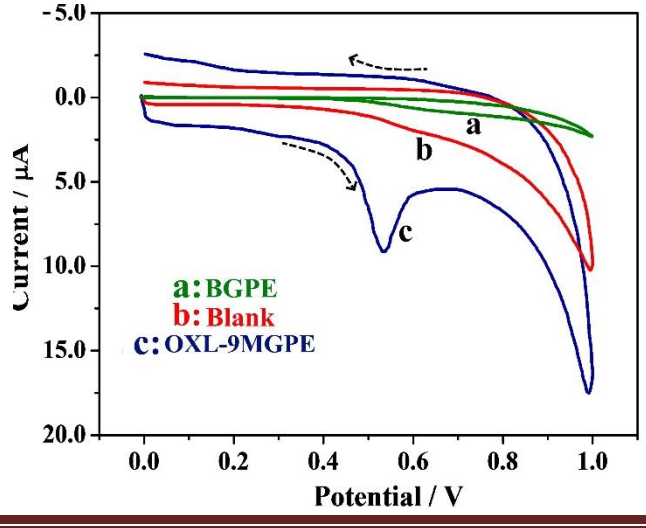
The study of the voltammetric analysis of the AC using the fabricated sensor was presented in this article. The voltammetric sensor based on the poly asparagine modified carbon nanotube and graphene mixed paste electrode (PASMCNTMGPE) was constructed. The constructed electrode unveiled a boosted electrocatalytic activity towards the detection of the AC than the unmodified electrode through the cyclic voltammetry (CV) and differential pulse voltammetry technique (DPV). FE-SEM was utilized to verify the morphological feature of the designed electrode. The charge transfer resistance of the designed electrode was studied by the electrochemical impedance spectroscopy (EIS). The redox reaction of the AC at the PASMCNTMGPE is pH-dependent and adsorption-controlled process. The designed electrode yielded the low detection limit (4.10×10^{-8} M) for AC. The PASMCNTMGPE displayed an outstanding selectivity. In addition, the prepared electrode demonstrated an excellent anti-fouling property, reliability and repeatability. More importantly, the feasibility of the constructed sensor was evaluated by analysing the AC in pharmaceutical and biological samples.



Chapter 6A: Surfactant Modified Graphite Paste Electrode as an Electrochemical Sensor for the Voltammetric Detection of Estriol with Dopamine and Uric acid

(**Advanced Pharmaceutical bulletin**, 2020, 10, 247-253,
https://doi.org/10.34172/apb.2020.029, ISSN: 2228-5881, **CiteScore: 4.8**)

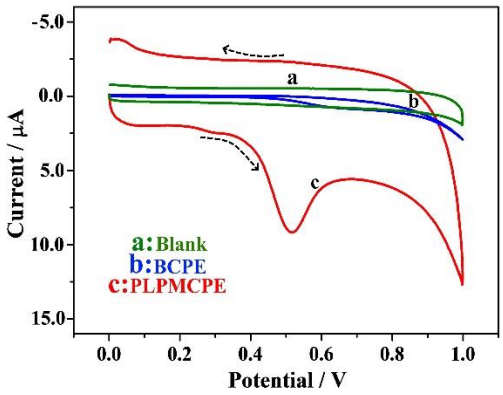
Estriol (ERL) is a type of hormone among the groups of estrogen hormone that was detected through the voltammetric technique by constructing an electrochemical sensor based on the Triton X-100 modified graphite paste electrode (TX-100MCPE). The fabricated electrode has substantial electrochemical sensing efficiency, and the ERL oxidation at the TX-100MCPE was the irreversible process. The surface morphological characteristics of BCPE and TX-100MCPE were differentiated with the help of FE-SEM. Techniques of CV and DPV were utilized to determine ERL, DAN, and URA simultaneously with the projected sensor. The detection limit 1.4×10^{-6} M and limit of quantification (LOQ) 4.7×10^{-6} M have been attained. More importantly, the prepared TX-100MCPE was successfully applied in the detection of the ET in the available ointment samples.



Chapter 6B: Poly (L-Proline) modified carbon paste electrode as the voltammetric sensor for the detection of Estriol and its simultaneous determination with Folic and Ascorbic acid

(Materials Science for Energy Technologies, 2, 2019, 365-371, https://doi.org/10.1016/j.mset.2019.05.002, ISSN: 2589-2991**)**

Electrochemical sensor based on electro-polymerization of L-Proline at the carbon paste electrode (CPE) by CV technique for the sensitive detection of Estriol (ET) was developed. The modification of CPE with poly (L-Proline) has enhanced the electrochemical oxidation of ET with increased current sensitivity in 0.1 M phosphate buffer solution (PBS) of pH 6.5 by CV and DPV. The surface morphology of both the bare carbon paste electrode (BCPE) and the modified carbon paste electrode (MCPE) was examined by utilizing FESEM. The linear range of 6×10^{-6}– 6×10^{-5} M with a limit of detection (LOD) 2.2×10^{-7} M and limit of quantification (LOQ) 7.6×10^{-7} M has obtained for ET.

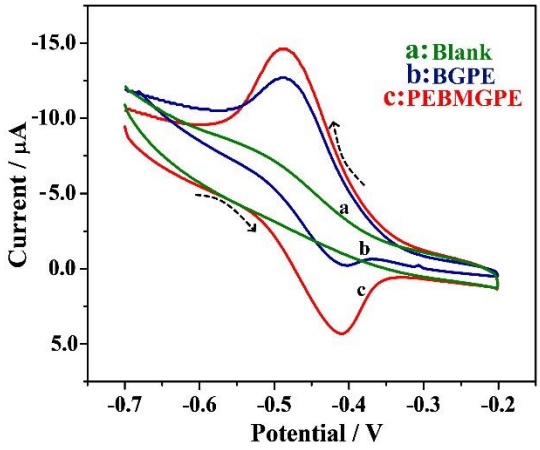


Chapter 7: Fabrication of Poly (Evans blue) Modified Graphite Paste Electrode as an Electrochemical Sensor for Sensitive and Instant Riboflavin Detection

(Moroccan Journal of Chemistry, 9, 2021, 007-017) https://doi.org/10.48317/IMIST.PRSM/morjchem-v9i1.18239. ISSN: 2351-812X)

Electro-analysis of Riboflavin (RN) consumed as vitamin has been delved by constructing the poly (Evans Blue) modified graphite paste electrode (PEBMGPE) as an electrochemical detector. Voltammetric determination of RN in 0.2 M PBS (pH 7.0) at the electro-active layer of the PEBMGPE was conducted. The PEBMGPE plays a crucial role as sensing tool towards the detection of RN compared to the BGPE. The topographical features of the bare graphite paste electrode (BGPE) and PEBMGPE was acquired by the FE-SEM. LOD and LOQ gained as 2.1×10^{-7} M and 7.1×10^{-7} M, respectively. Subsequently, good repeatability, excellent reproducibility, and adequate stability, high sensitivity were achieved for the

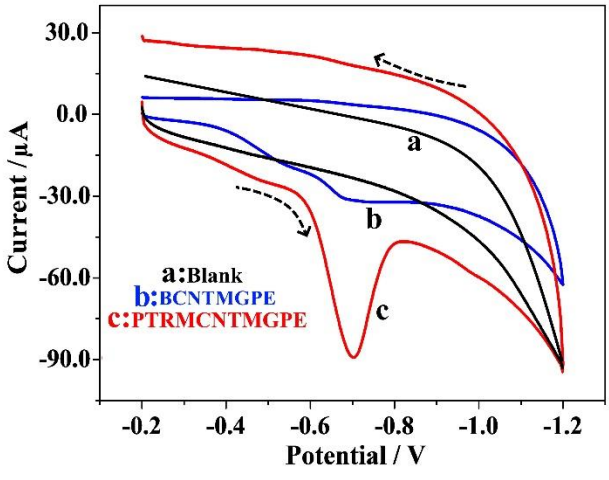
PEBMGPE sensor. Besides, the practicability of the projected sensor was utilized for the detection of the RN in pharmaceutical formulations.



Chapter 8: Electroanalytical Determination of Tinidazole using surface modified carbon Nano composite based sensor

(Materials Research Innovations, 2021 https://doi.org/10.1080/14328917.2021.1969723, ISSN 1432-8917, CiteScore: 3.1**)**

The electrochemical sensor based on poly threonine modified carbon nanotube mixed graphene paste electrode (PTRMCNTMGPE) was developed. The modified electrode displayed a boosted electrocatalytic effect towards the voltammetric signal of the TNZ compared to the bare electrode. The morphology and charge transfer resistance of the proposed electrodes were characterised by FE-SEM and EIS. The scan rate study reveals that the reduction reaction of TNZ at the modified electrode is controlled by diffusion. The designed electrode yielded low detection limit for TNZ. The proposed electrode exhibited a good reproducibility, repeatability and an antifouling feature. The practicability of the developed sensor was explored by detecting the TNZ in table and blood serum samples.



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LIST OF PUBLICTIONS:

Paper Published/Accepted/Communicated in national/international journals

1. **Charithra M. M**, Manjunatha J. G, Electrochemical sensing of adrenaline using surface modified carbon nanotube paste electrode. (**Materials Chemistry and Physics**, 262, 2021, 124293, https://doi.org/10.1016/j.matchemphys.2021.124293 ISSN: 0254-0584, **I. F: 4.0**)
2. **Charithra M. M,** Manjunatha J. G, Polymerized Carbon Nanotube Paste Electrode as a Sensing Material for the Detection of Adrenaline with Folic acid (Accepted by **Monatshefte für Chemie - Chemical Monthly**, ISSN: 0026-9247, **I. F: 1.45**)
3. **Charithra M. M**, Manjunatha J. G, Electrochemical Sensing of Paracetamol Using Electropolymerised and Sodium Lauryl Sulfate Modified Carbon Nanotube Paste Electrode (**ChemistrySelect,** 5, 2020, 9323-9329, 10.1002/slct.202002626, ISSN: 2365-6549. **I. F. 2.01**)
4. **Charithra M. M**, Manjunatha J. G, Electroanalytical Determination of Acetaminophen using polymerised Carbon Nanotube Based Sensor (Accepted in **Journal of Electronic Materials,** ISSN: 0361-5235, **I. F. 1.8**)
5. **Charithra M. M,** Manjunatha J. G, Enhanced voltammetric detection of paracetamol by using carbon nanotube modified electrode as an electrochemical sensor (**Journal of Electrochemical Science and Engineering,** 10, 2020, 29-40, http://dx.doi.org/10.5599/jese.717. ISSN 1847-9286, **CiteScore: 1.7**)
6. **Charithra M. M**, Manjunatha J. G, Electroanalytical Determination of Acetaminophen using Polymerized Carbon Nanocomposite based Sensor (**Chemical Data Collections**, 33, 2021, 100718, https://doi.org/10.1016/j.cdc.2021.100718, ISSN: 24058300, **CiteScore: 1.2**)
7. **Charithra M. M**, Manjunatha J. G, Surfactant Modified Graphite Paste Electrode as an Electrochemical Sensor for the Enhanced Voltammetric Detection of Estriol with Dopamine and Uric acid. (**Advanced Pharmaceutical Bulletin**, 2020, 10(2), 247-253 doi: 10.34172/apb.2020.029. **citeScore: 4.8**)
8. **Charithra M. M**, Manjunatha J. G, Poly (L-Proline) modified carbon paste electrode as the voltammetric sensor for the detection of Estriol and its simultaneous determination with Folic and Ascorbic acid. (**Materials Science for Energy Technologies** 2, 2019, 365-371. https://doi.org/10.1016/j.mset.2019.05.002)

9. **Charithra M. M**, Manjunatha J. G, Fabrication of Poly (Evans blue) Modified Graphite Paste Electrode as an Electrochemical Sensor for Sensitive and Instant Riboflavin Detection. (**Moroccan Journal of Chemistry**, 9, 2021. https://doi.org/10.48317/IMIST.PRSM/morjchem-v9i1.18239. ISSN: 2351-812X)
10. **Charithra M. M,** Manjunatha J. G, Sensitive Detection of Anthrone using Poly (L-Arginine) modified Carbon paste electrode by Voltammetric Method (**Open Access Journal of Chemistry**, 3, 2019, 8-14, ISSN: 2624-8549)
11. **Charithra M. M**, Manjunatha J. G, Electroanalytical Determination of Tinidazole using surface modified carbon Nano composite based sensor (**Materials Research Innovations, 2021** https://doi.org/10.1080/14328917.2021.1969723, ISSN 1432-8917, CiteScore: 3.1)
12. **Charithra M. M**, Manjunatha J. G, Voltammetric Detection of Amoxicillin by using Poly (l-Aspartic acid) modified carbon paste electrode (Communicated to Iraninan Chemical Society, **I. F. 2.0**)
13. **Charithra M. M**, Manjunatha J. G, Electroanalysis of Epinephrine by using poly (l-asparagine) modified carbon Nanocomposite electrode (Communicated to **Physical Chemistry Research**, ISSN 2345-2625).
14. Manjunatha J. G, Raril C, Hareesha N, **Charithra M. M**, Pushpanjali P. A, Girish T, Ravishankar D. K, Mallappaji S. C, Jayarame G**,** Electrochemical Fabrication of Poly (niacin) Modified Graphite Paste Electrode and its Application for the Detection of Riboflavin. (**The open chemical engineering,** 14, 2020 DOI: 10.2174/1874123102014010090. **I. F.: 0.46**)
15. Hareesha N, Manjunatha J.G, Amrutha B.M, Pushpanjali PA, **Charithra M.M**, Prinith Subbaiah N, Electrochemical Analysis of Indigo Carmine in Food and Water Samples Using a Poly (Glutamic Acid) Layered Multi-walled Carbon Nanotube Paste Electrode. (**Journal of Electronic Materials**, 50, 2021, 1230-1238, https://doi.org/10.1007/s11664-020-08616-7. **I. F.: 1.6)**

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1. **Charithra M.M,** Manjunatha J. G, Voltammetric detection of Paracetamol by using Carbon Nanotube Modified electrode as an Electrochemical Sensor. International conference on electrochemistry in industry, health and environment (EIHE-2020) held at Bhabha Atomic Research Center (BARC), January 21-25th at Mumbai.2020.

2. **Charithra M.M,** Manjunatha J. G, Poly (Evans Blue) modified graphite paste electrode as a potential electrochemical sensor for the Sensitive and Instant Riboflavin Detection. International Conference on Advances in Chemical & Materials Sciences (ICCM), Department of Studies in Chemistry, Mangalore University, 17-19 October, 2019.
3. **Charithra M.M,** Manjunatha J. G, Surfactant Modified Graphite Paste Electrode as an Electrochemical Sensor for the Enhanced Voltammetric Detection of Estriol with Dopamine and Uric acid. Department of Chemistry, JSS Science & Technology University, Mysuru, National Conference on "Advanced Materials for Health, Energy and Environment", 6-7 September, 2019.
4. **Charithra M.M,** Manjunatha J. G, Surfactant Modified Graphite Paste Electrode as an Electrochemical Sensor for the Enhanced Voltammetric Detection of Estriol with Dopamine and Uric acid. International Conference on "Current concepts on the role of Indian medicine and phytoceuticals in maintainance of health", PG Centre, Chikk Aluvara, Mangalore University, 15-17 November, 2018.
5. **Charithra M. M,** Research Methodology one day work shop, Field Marshal KM Cariappa College, Madikeri, Karnataka, 19th April, 2018.
6. **Charithra M. M,** Manjunatha J. G, Surfactant Modified Graphite Paste Electrode as an Electrochemical Sensor for the Enhanced Voltammetric Detection of Estriol with Dopamine and Uric acid, FMKMC College Madikeri, Mangalore University Constituent College, 5th February 2019.
7. **Charithra M. M,** Manjunatha J. G, Poly (L-Proline) modified carbon paste electrode as the voltammetric sensor for the detection of Estriol and its simultaneous determination with Folic and Ascorbic acid, FMKMC College Madikeri, Mangalore University Constituent College, 12th February 2020.

Research Scholar — Research Guide

(Charithra M. M) — (Dr. J. G. Manjunatha)